

HYDROGENATION OF COAL TAR
AND COAL TAR OILS

BY

R. S. MELICHAR
J. ZAVERTNIK, JR.

ARMOUR INSTITUTE OF TECHNOLOGY

1919

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The hydrogenation of coal
tar and coal tar oils



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THE HYDROGENATION OF COAL TAR AND COAL TAR OILS

A THESIS

PRESENTED BY

ROBERT S. MELICHAR

TO THE

PRESIDENT AND FACULTY

OF

ARMOUR INSTITUTE OF TECHNOLOGY

FOR THE DEGREE OF

BACHELOR OF SCIENCE

IN

CHEMICAL ENGINEERING

MAY 29, 1919

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THE HYDROGENATION OF COAL TAR AND COAL TAR OILS

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INTRODUCTION.

Three problems present themselves to the tar distiller today. These are:-

In distilling tar for its products, fifty to seventy per cent of the tar is obtained as pitch. The outlook for pitch is exceedingly problematical. There is a possible outlet in the fuel field but competition here is too great. The production of tar has increased 100% in the last few years.

Tar oils which are liquid at ordinary temperatures are in much greater demand than tar oils which solidify at ordinary temperatures. The latter oils contain naphthalene which is the main constituent that causes them to solidify.

There is practically no demand for naphthalene. During the war, a considerable amount of it was used by the Ordnance Department of the U.S. Government. An outlet is therefore sought for the naphthalene which constitutes 5 to 7% of the tar.

By passing hydrogen through the tar

during distillation, it was thought that a larger production of oil could be obtained per gallon of tar. The idea being that the hot gas would be capable of absorbing the products of distillation and thus carry more of them over.

It was also thought that by passing the hydrogen through the tar, a circulation of the tar in the still would be maintained and no local overheating would take place and thus the "free carbon" content of the pitch would be kept down to a minimum. This would tend to reduce the decomposition or "cracking" of the tar and thus increase the oil yield.

By passing hydrogen through the tar, an intimate mixture of the oils and hydrogen would be obtained. Hydrogenation would then take place in the "Contact Chamber" which would contain the catalytic agent and possibly change the constituents of the distillates to produce a more liquid oil.

ABSTRACT.

Tests were conducted to determine the effect Hydrogen has on Coal Tar and its distillates during the process of distillation.

Heated Hydrogen was passed through the Coal Tar during distillation. The mixed vapors were allowed to come in contact with the catalytic agents at a temperature above the boiling point of the products. The catalytic agents used were aluminum chloride and nickle. Factor studied was the catalytic agent at atmospheric pressure. It would be of interest to have further work done to study the following factors, Coal Tar, temperature of the "Contact Chamber", pressure of the system, Vacuum, atmospheric and above atmospheric, and various other catalytic agents.

From tests conducted would conclude that polymerization has taken place, as is evident by the increase of anthracene salts obtained in the total oil when the catalytic agents were used. Carbon and uncondensable gas are

obtained either as products of decomposition
or of polymerization.

PRELIMINARY OUTLINE OF WORK.

OBJECT: To determine the effect hydrogen has upon Coal Tar and its distillates during the process of Distillation; and the percentage of oil yield per gallon of Coal Tar when hydrogenation takes place.

OUTLINE OF PROCEDURE: Heated Hydrogen is to be passed through the Coal Tar during the process of distillation. The mixed vapors are to be allowed to come in contact with a catalytic agent at a temperature above the boiling point of the products.

VARIABLE FACTORS TO BE STUDIED:

1. Coal Tar.
2. Catalytic Agent.
3. Temperature of Entering Hydrogen.
4. Temperature of the "Contact Chamber".
5. Pressure of the System.
 - (a) Vacuum.
 - (b) Atmospheric.
 - (c) Above Atmospheric.

DATA TO BE OBTAINED ON EACH RUN:

% Water in Crude Tar.
% Light Oil Dist. to 205° C.
% Heavy Oil Dist. to 140° F. Pitch.
Final Temp. of Distillation.
M.P. of Pitch. (Water)
% Heavy Oil Dist. to 300° F. Pitch.
Final Temp. of Distillation.

M.P. of Pitch (Air).
 Temperature of Hydrogen entering
 Still.
 Specific Gravity of dry Tar @ 60⁰ F.
 % of Dry Tar Insoluble in Benzol.
 % Ash on dry tar.
 Viscosity (Engler) 100_{cc} @ 50⁰ C.
 Viscosity (Engler) 100_{cc} @ 115⁰ C.

TEST OF HEAVY OIL (140⁰ F Pitch)

Specified Gravity @ 15.5⁰ C.
 % Naphthalene.
 Limpid Point.
 % Anthracene Salts.
 Dry Solids @ 40⁰ F. (4⁰ C)
 % Tar Acids.
 % Unsulphonated Residue.

	% to 170 ⁰ C.
Standard	% to 200 ⁰ C.
Retort	% to 210 ⁰ C.
Distilla-	% to 235 ⁰ C.
tion	% to 315 ⁰ C.
A.S.T.M.	% to 355 ⁰ C.
D.-7.	

Sp. Gr. Fraction (235⁰ C-315⁰ C.)
 @ 38⁰ C/15.5⁰ C.

TEST OF HEAVY OIL (140⁰ F. Pitch) (to 300⁰ F.A.B.) (Pitch)

Specific Gravity @ 15.5⁰ C.
 Limpid Point.
 % Naphthalene.
 % Anthracene Salts.
 Dry Solids @ 40⁰ F (4⁰ C)
 % Tar Acids.
 % Unsulphonated Residue.

	% to 170 ⁰ C.
Standard	% to 200 ⁰ C.

Retort	%	to	210 ⁰	C.
Distill-	%	to	235 ⁰	C.
ation	%	to	315 ⁰	C.
A.S.T.M.	%	to	355 ⁰	C.

Sp. Gr. Fraction (235⁰ C-315⁰ C.
 @ 38 C/15.5 C.

TEST OF HEAVY OIL (205⁰ to 300⁰ F. A.E. Pitch)

Specific Gravity @ 15.5⁰ C.

Limpid Point.

% Naphthalene.

% Anthracene Salts.

Dry Solids @ 40⁰ F (4⁰ C)

% Tar Acids.

% Unsulphonated Residue.

	%	to	170 ⁰	C.
Standard	%	to	200 ⁰	C.
Retort	%	to	210 ⁰	C.
Distill-	%	to	235 ⁰	C.
ation	%	to	315 ⁰	C.
A.S.T.M.	%	to	355 ⁰	C.
D-7.	%	to	355 ⁰	C.

Sp. Gr. Fraction 235⁰ C-315⁰ C.
 @ 38⁰ C/15.5⁰ C.

% Insoluble in Benzol of 140⁰ F Pitch.

% Insoluble in Benzol of 300⁰ F Pitch:

Melting Point of Bitumen of 300⁰ F
 Pitch.

% of Total oil yield.

% of 300⁰ F. Pitch.

% to Distillation Loss or Gain.

So far as the writers know, no work has been done on the hydrogenation of Coal Tar and its distillates during the process of distillation. The literature only discusses hydrogenation of aromatic hydrocarbons in a pure state. Ipatiev hydrogenates benzene derivatives by passing them with dry hydrogen over iron, copper or aluminum at a temperature of 400 to 450⁰ C. Sabatier and Senderens showed that at 160⁰ C., benzene is hydrogenized by catalysts, such as finely divided nickel, etc., to cyclo-hexane, and that by passing cyclo-hexane over the same materials at 270 to 280⁰ C., it is reduced to benzene. Smith and Lewcock, have passed benzene through an iron tube heated from 600 to 800⁰ C., and various oxides were used as catalysts, but they soon became coated with carbon. Yields up to 59% diphenyl were obtained. Ubbelohde, St. Philippide, Woronin, Phillips, Bulteel, Day, Hall and Holegreber use hydrogen and a catalytic agent to hydrogenate the Petroleum or its distillates to produce light oils

from the heavier product. The literature discusses to a great extent the hydrogenation of Petroleum, aliphatic hydrocarbon, and only to a very limited extent the hydrogenation of aromatic hydrocarbons.

DESCRIPTION OF RUNS.

RUN 1.

A still (20 c.m. in diameter and 30 cm. high) with a cubical content 9500 c.c. was charged with 7000 gm. of tar. It was heated by means of two ring burners (one at about the level of the tar and the other at the level of the sampling cock which was about 5 or 6 cm. from the bottom) and a rose burner. The still was connected to an air condenser by means of a Hempel distilling tube containing a thermometer. The air condenser delivered into a bottle from which any uncondensed vapors were led into a water condenser.

All joints and possible leaks were plastered up by the following method. A mixture of plaster of paris and asbestos (made up to a putty) was applied to the joints. When this hardened a paste of plaster of paris and glycerine was applied and lastly, if necessary, a coat of water glass.

Run 1 was a regular distillation as is

used to analyze tar (complete tar test). In starting, only the top ring burner was lighted at a low flame and the heat gradually increased. When more heat was required the bottom burner was started and finally, the rose was also used to finish the distillation.

The data and tests obtained are shown in tabulated form. 1,620 c.c. of oil at 60⁰ F, were obtained. The melting point of pitch was 143⁰ F (water) and the vapor temperature was 315⁰ C when the first cut was made. The distillation was carried on and 1530 c.c. of oil at 60⁰ F were obtained, for the second cut. The melting point of pitch was 412⁰ F (AB) and the vapor temperature was 420⁰ C.

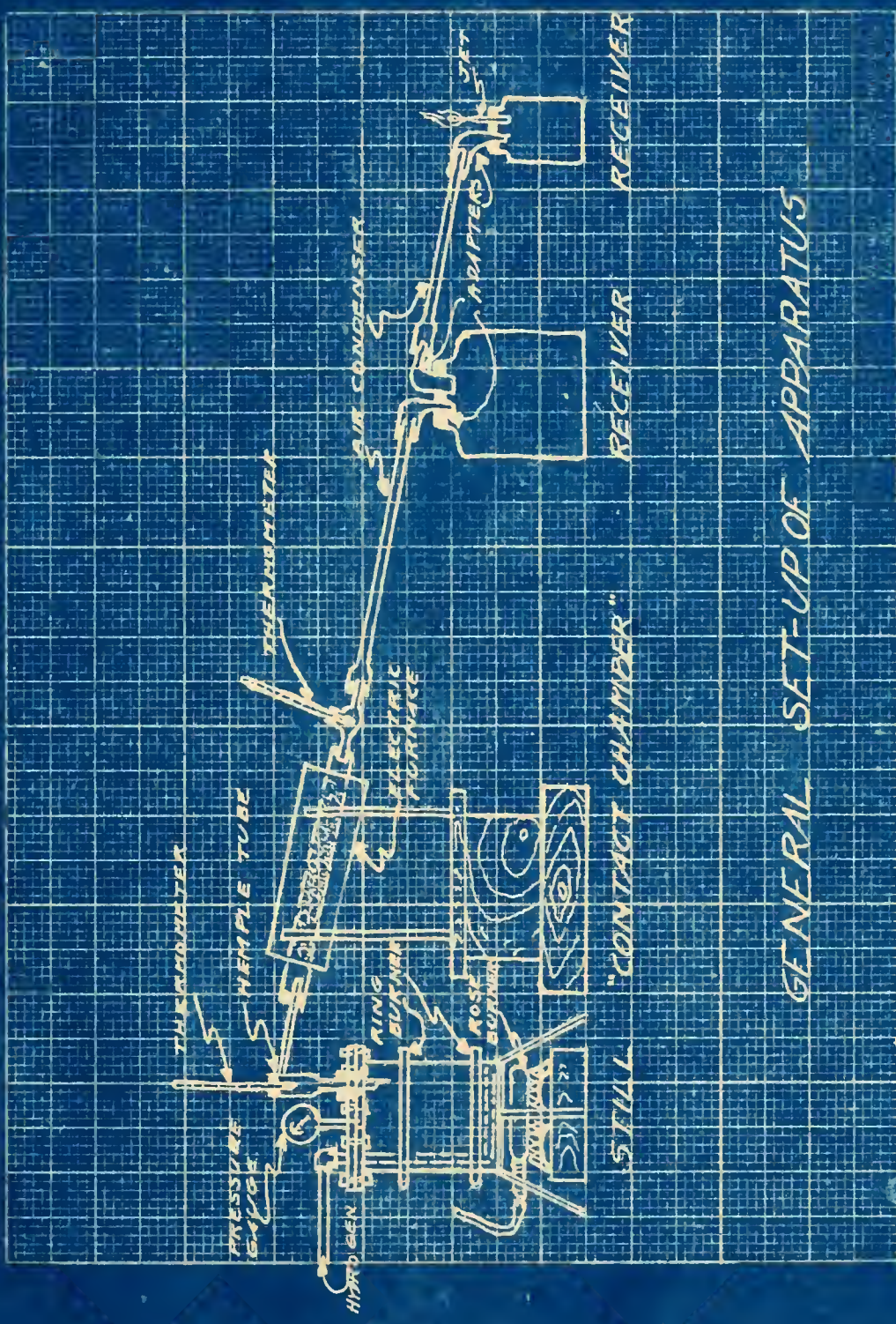
RUN 2.

The apparatus was set up as in Run 1 with the following changes. The receiver at the end of the water cooled condenser was enclosed by a bell glass with open top, with tabulature near bottom and with wide well ground flange. A motor driven vacuum pump was connected to the bell glass by means of the tabulature. It was very dif-

ficult to maintain a vacuum. Leaks were stopped in a similar manner as in Run 1. An attempt was made to carry a high vacuum of about 24" but it was abandoned on account of too much "foaming". A vacuum of 17" was maintained during the process of distillation. Leaks and other trouble made it impossible to complete this distillation. The data obtained was as follows: 1860 c.c. at 60° F. (water) melting point pitch and a vapor temperature of 300° C. Tests are embodied in table.

RUN 3.

Run 3 was the first hydrogenation run. Hydrogen (as obtained in a tank from the Burdette Oxygen Co.,) was washed first with sulphuric acid and then with caustic soda. Regular ground glass stoppered wash bottles were tried but they proved unsuccessful. The slight pressure of the system blew off the stoppers and produced considerable leakage. Ordinary wide mouth bottles with tight rubber stoppers which were wired on proved very successful. The glass tubing that extended into



GENERAL SET-UP OF APPARATUS

the bottle thru the stopper was forced thru it after the stopper was in place. Of course, care being taken not to break the tubing. This made an air tight stopper.

The hydrogen was led from the washing bottles thru a 1/4" check valve to a 1/4" iron pipe 2 1/2' ft. long which was heated by two wing top Bunsen burners. The pipe was continued into the still being screwed thru the top of it and terminated at the bottom of the still in a perforated ring of 1/4" iron piping. The outside diam. of the ring was about 1" smaller than the inside diam. of the still.

A Hempel distilling tube containing a thermometer led the vapors and hydrogen to the "Contact Chamber". This was a silica tube (1" diam. and 2' long) containing the catalytic material and heated by a standard 110 volt, 5 ampere electric furnace. The catalytic material was made by dissolving 50 grms. of aluminum chloride in water. Asbestos fiber was soaked in this solution for about 12 hrs. and the whole evaporated to dryness. This material was placed in the

silica tube, loosely packed, so as to permit the distillates to pass thru.

This silica tube was joined to a T - tube made of 1/2" glass tubing and with a bulb at the junction of the legs. The bulb gave ample room for a thermometer bulb to be placed in it without forming too great an obstruction to the stream of distillate. The temperature of the exit vapors and gas was kept about 60⁰ C above the temperature of vapors and gas leaving the still.

This T tube was followed by two air condensers. The water condenser used in previous runs had to be discarded because it was being continually plugged with solid distillate and no flame could be applied to it to melt same. As before, a bottle receiver connected the two condensers. The end receiver i. e. the receiver at the end of the 2nd air condenser, was supplied with a glass jet for burning the excess hydrogen and uncondensable gas. The gases coming off were not analyzed, but due to the luminosity of the flame one would be led to assert that a gas other than pure hydro -

gen was being burned; probably hydrocarbons mixed with hydrogen.

Data obtained: 1842 c.c. at 60⁰ F to 147⁰ F (water) melting point pitch was distilled. Final temperature of distillation was 310⁰ C. Tests in table.

Due to an accident, the distillation had to be discontinued. The catalytic material used in the contact chamber swelled considerably, and had a metallic luster, shown that aluminum chloride had been reduced to the metal aluminum. Carbon was deposited on it to such an extent that no distillate could possibly pass thru. A hydrogen flame was burning to the end. It was not anticipated by the workers that it was possible for hydrogen to pass thru the plug while the distillate was held back. As a result of the pressure which was built up in the still by the accumulated vapors of distillation((the check valve on the entrance pipe prevented the back pressure to force the contents of the still back into the wash bottles), the contents of the still was blown out thru the Hempel tube and ignited, that

being the weakest point and the line of least resistance. It was therefore decided in subsequent runs to connect a pressure gauge to the still to indicate an increased pressure if it occurred and pumice stone was substituted for asbestos fiber.

RUN 4.

Except for the catalytic material and the pressure gauge Run 4 was identical to Run 3. About 100 grms. of nickel nitrate was dissolved in water and pieces of pumice stone were left to soak in it for 20 hrs. After this, the whole was evaporated to dryness. The pieces of pumice were then ignited giving nickelic oxide in a finely divided state in the pumice. The tube of the furnace was then packed loosely with this material and the distillation carried on as in the previous run.

Data obtained: 1671 c.c. at 60° F. to 130° F (water) melting point pitch were distilled with final temperature of 395° C. Second cut was 1716 c.c. at 60° F to 305° F (air) melting point pitch with final temperature of distillation of



428⁰ C.

RUN 5.

This run was made with 100 grms. of anhydrous aluminum chloride in still and aluminum chloride (prepared with pumice) in the "Contact Chamber". Other conditions were identical to previous runs.

Data obtained : Light Oil to 162⁰ F (water) pitch at 60⁰ F - 1615 c.c. Final temperature of cut 370⁰ C. Heavy Oil from 162⁰ F (water) pitch to 391⁰ F (air) pitch at 60⁰ F 1379 c.c. Final temperature of distillation 391⁰ C. Other tests in table.

RUN 6.

To ascertain whether the aluminum chloride in the still or in the "Contact Chamber" produced a change in the distillates, it was deemed necessary to make a run with aluminum chloride in the still, and no catalytic material in the "Contact Chamber". This was the only difference between Run 6 and previous runs.

Data obtained : Light Oil to 153° F (water) pitch at 60° F 1408 c.c. Final temperature of cut 314° C. Heavy Oil from 153° F (water) pitch to 342° F (air) pitch at 60° F 1553 c.c. Final temperature of distillation 425° C. Other tests in table.

RUN 7.

As a means of checking up, Run 7 was made without passing hydrogen thru the system and no catalytic material in the "Contact Chamber" or in the still; other conditions were the same as in the other runs.

Data obtained: Light Oil to 137° F (water) pitch at 60° F - 1575 c.c. Final temperature of cut 365° C. Heavy oil from 137° F (water) pitch to 378° F (air) 1704 c.c. at 60° F. Final temperature of distillation 445° c.c. Other tests in table.



<u>TEST OF TAR</u> <u>RUN NO.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
% Water	0.0	0.0	0.0	0.0	0.0	0.0	0.0
C.C.Light Oil Dist. to 140°F. Pitch at 60°F.	1620	1860	1842	1671	1615	1408	1575
% Light Oil Dist. to 140°F. Pitch-Fract.A (by vol.)	27.7	31.8	31.7	28.5	27.6	24.1	26.9
Final Temp.of Distillation	315°C.	300	310	395	370	314	365
M.P.of Pitch (Water)	143	153	147	139	162	153	137
C.C.Hy.Oil Dist. from 140°F. Pitch to 300°F.Pitch at 60°F-1530	-	-	-	1716	1379	1553	1704
% Heavy Oil Dist. from 140°F.Pitch to 300°F.Pitch ØFract.B) by volume	26.1	-	-	29.3	23.7	26.6	29.2
C.C.Heavy Oil Dist. to 300°F.Pitch by volume at 60°F.	3150	-	-	3387	2994	2961	3279
% Heavy Oil Dist. to 300°F. Pitch (Fract.C) by volume	53.8	-	-	57.8	51.3	50.7	56.1

TEST OF TAR

<u>RUN NO.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
Final Temp. of Distillation	420°C.	-	-	428	445	425	445
M.P. of Pitch (Air) - °F.	412	-	-	405	391	342	378

TEST OF DRY TAR

Spec. Grav. at 60°F.	1.196	1.196	1.196	1.196	1.196	1.196	1.196
% Insoluble in Benzol	10.37	10.37	10.37	10.37	10.37	10.37	10.37
% Ash	0.15	0.15	0.15	0.15	0.15	0.15	0.15
Viscosity (Engler) 100cc. @ 50°C	314	314	314	314	314	314	314
"	"	115 "	32	32	32	32	32

TEST OF FRACTION A - LIGHT OIL DIST. TO 140°F. PITCH

Specific Gravity @ 15.5°C.	1.043	1.054	1.063	1.056	1.077	1.067	1.061
Limpid Point	108	101	103	103	101	106	105
% of Solid up to 200°C.	0.4	0.6	0.5	0.9	0.4	2.5	0.8

The first of these is the fact that the
 number of cases of the disease has
 increased in the last few years.
 This is due to the fact that the
 disease is more common in the
 tropics than in the temperate
 regions.

The second fact is that the
 disease is more common in the
 tropics than in the temperate
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The fifth fact is that the
 disease is more common in the
 tropics than in the temperate
 regions. This is due to the fact
 that the disease is more common
 in the tropics than in the
 temperate regions.

TEST OF FRACTION A (Cont'd)

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
% of Naphthalene (Fract. 200-250°C.)	26.3	22.0	22.0	24.0	19.0	24.2	23.4
% of Solids (Fract.250- 300°C.)	1.3	0.8	1.9	0.8	1.4	0.2	0.3
% of Anthracene Salts (Fract.300-390°C.)	3.3	5.4	6.5	5.3	6.6	4.3	5.0
Dry Solids	21.2	16.3	20.8	20.8	20.0	21.3	22.6
% Tar Acids	6.8	6.8	6.0	6.0	5.0	6.5	6.8
% Unsulphonated Residue	1.0	1.0	0.2	0.2	0.2	0.2	0.2
STANDARD RETORT	0.3	0.2	0.0	0.4	0.0	0.0	0.0
DISTILLATION	3.0	1.2	1.0	1.4	1.1	2.5	0.8
A.S.T.M.	5.0	2.7	2.6	3.3	3.8	5.0	2.5
D-7	47.7	45.5	36.8	37.8	30.8	42.8	37.4
	88.3	84.3	79.2	80.5	72.0	76.2	62.8
	97.0	94.5	94.0	94.0	88.3	90.6	85.8

TEST OF FRACTION A (Cont'd)

Spec. Grav. Fraction 235-315°C
at 38°C/15.5°C.

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
	1.036	1.035	1.039	1.037	1.038	1.036	1.035
(% to 170°C.)	1.5	0.0	0.0	0.0	3.0	3.0	0.0
(% " 200 ")	3.5	2.1	3.5	3.0	6.0	10.0	2.0
(% " 210 ")	9.0	8.1	10.0	9.0	10.0	19.0	8.0
(% " 235 ")	49.0	46.8	42.0	47.0	37.0	52.0	39.0
(% " 250 ")	66.0	62.3	54.0	61.5	52.5	65.0	59.5
(% " 300 ")	84.0	78.5	75.0	78.0	66.5	80.0	73.0
(% " 315 ")	89.5	86.4	81.0	81.0	73.0	85.0	75.0
(% " 355 ")	(95.0) (340°C)	(96.7) (340°C)	91.0	91.5	86.0	92.0	83.0
(% " 390 ")	-	-	(95.0) (370°C)	(96.0) (380°C)	90.0	94.0	90.0

TEST OF FRACTION B - HEAVY OIL
DIST. FROM 140°F. Pitch 300°F.A.B.PITCH

Specific Gravity @15.5°C.	1.145	-	-	1.142	1.142	1.145	1.148
Limpid Point °F.	136	-	-	126	142	140	137

<u>TEST OF FRACTION B (Cont'd)</u>							
	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
% Solids in fraction up to 200°C.	0.0	-	-	0.0	0.0	0.0	0.0
% of Naphthalene Fract. 200-250°C.	0.0	-	-	0.0	0.0	0.2	0.3
% of Solids fraction (250-300°C.)	0.3	-	-	0.2	0.2	0.2	0.3
% of Anthracene Salts (300-390°C.)	9.5	-	-	9.2	10.0	7.5	6.3
Dry Solids	9.5	-	-	9.4	11.0	6.8	5.3
% Tar Acids	2.2	-	-	2.8	2.0	2.0	4.0
% Unsulphonated Residue	0.2	-	-	0.0	0.0	0.0	0.0
STANDARD	0.0	-	-	0.0	0.0	0.0	0.0
RETORT	0.0	-	-	0.0	0.0	0.5	0.0
DISTILLATION	0.0	-	-	0.2	0.0	1.0	0.0
A.S.T.M.	0.0	-	-	0.9	0.0	2.0	0.8
D-7	3.0	-	-	9.8	7.0	20.0	8.0
	49.6	-	-	41.8	36.2	51.8	33.2
	75.0	-	-	68.0	71.4	73.5	60.0

Spec. Grav. Fract. 235-315°C.
at 38°C/15.5°C.

	1	2	3	4	5	6	7
	Not sufficient		Not sufficient				
BULB							
DISTILLATION	(% to 170°C.)	0.0	-	0.0	0.0	2.0	0.0
	(% " 200 ")	0.0	-	1st Drop	0.0	2.5	0.0
	(% " 210 ")	0.0	-	1.0	0.0	3.5	0.0
	(% " 235 ")	0.0	-	2.0	1.5	6.0	3.0
	(% " 250 ")	1.0	-	3.0	2.5	8.0	5.0
	(% " 300 ")	7.0	-	8.0	9.0	20.0	14.0
	(% " 315 ")	12.5	-	14.0	15.0	25.5	20.0
	(% " 355 ")	44.5	-	40.0	41.0	52.5	38.0
	(% " 390 ")	73.0	-	68.0	71.0	74.0	58.0
				435°F-95.0			

TEST OF FRACTION C
HEAVY OIL DIST. TO 300°F. (A.B. PITCH)

Specific Gravity @15.5°C.	1.090	-	-	1.099	1.109	1.107	1.107
Limpid Point	108	-	-	136	135	137	133
% Solids in fraction up to 200°C.	0.4	-	0	0.4	0.2	0.3	0.2
% Naphthalene (fraction 200-250°C.)	10.5	-	-	10.5	6.4	10.3	8.5

TEST OF FRACTION C (Cont'd)

	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>	<u>7</u>
% Solids (fraction 250-300°C.)	0.5	-	-	0.2	3.2	2.3	2.8
% of Anthracene Salts (300-390°C.)	6.6	-	-	7.5	8.3	6.3	5.0
Dry Solids	8.0	-	-	7.0	3.5	5.8	3.8
% Tar Acids	3.2	-	-	4.5	3.6	4.0	5.5
% Unsulphonated Residue	0.6	-	-	0.1	0.1	0.1	0.1
STANDARD RETORT DISTILLATION A.S.T.M. D-7	(% to 170°C.) (% " 200 ") (% " 210 ") (% " 235 ") (% " 315 ") (% " 355 ") (% " 390 ")	- - - - - - -	- - - - - - -	0.0 0.7 2.1 12.6 45.6 71.2 -	0.0 0.4 0.8 6.7 40.0 61.2 81.7	0.0 1.0 2.4 15.0 48.0 71.4 -	0.0 0.2 1.1 8.6 39.9 62.4 73.8
Spec. Grav. Fraction 235-315°C at 38°C/15.5°C.	1.037	-	-	1.032	1.038	1.037	1.038

TEST OF FRADTION C(Cont'd)

	1	2	3	4	5	6	7
BULB	0.0	-	-	0.0	0.0	1.0	0.0
DISTILLATION	4.0	-	-	3.0	1.0	5.0	2.0
(% to 170°C.)	5.0	-	-	5.0	4.5	9.0	5.0
(% " 200 ")	18.0	-	-	19.0	12.0	20.5	18.0
(% " 210 ")	28.5	-	-	28.0	21.0	30.0	27.0
(% " 235 ")	47.0	-	-	42.5	40.0	45.0	42.0
(% " 250 ")	51.5	-	-	46.5	45.5	49.5	46.0
(% " 300 ")	72.0	-	-	65.0	63.0	68.0	63.0
(% " 315 ")	87.0	-	-	78.0	79.0	81.0	77.0
(% " 355 ")							
(% " 390 ")							

TEST OF PITCH

Melting Point of Pitch (Water) °F.	143	153	147	139	162	153	137
% Insoluble in Benzol	17.38	16.01	18.34	18.83	19.63	18.21	16.98
Melting Point of Pitch (Air Bath °F.)	412	-	-	405	391	342	378
% Insoluble in Benzol	56.34	-	-	49.99	53.63	45.48	48.36

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89	90	91	92	93	94	95	96	97	98	99	100	101	102	103	104	105	106	107	108	109	110	111	112	113	114	115	116	117	118	119	120	121	122	123	124	125	126	127	128	129	130	131	132	133	134	135	136	137	138	139	140	141	142	143	144	145	146	147	148	149	150	151	152	153	154	155	156	157	158	159	160	161	162	163	164	165	166	167	168	169	170	171	172	173	174	175	176	177	178	179	180	181	182	183	184	185	186	187	188	189	190	191	192	193	194	195	196	197	198	199	200	201	202	203	204	205	206	207	208	209	210	211	212	213	214	215	216	217	218	219	220	221	222	223	224	225	226	227	228	229	230	231	232	233	234	235	236	237	238	239	240	241	242	243	244	245	246	247	248	249	250	251	252	253	254	255	256	257	258	259	260	261	262	263	264	265	266	267	268	269	270	271	272	273	274	275	276	277	278	279	280	281	282	283	284	285	286	287	288	289	290	291	292	293	294	295	296	297	298	299	300	301	302	303	304	305	306	307	308	309	310	311	312	313	314	315	316	317	318	319	320	321	322	323	324	325	326	327	328	329	330	331	332	333	334	335	336	337	338	339	340	341	342	343	344	345	346	347	348	349	350	351	352	353	354	355	356	357	358	359	360	361	362	363	364	365	366	367	368	369	370	371	372	373	374	375	376	377	378	379	380	381	382	383	384	385	386	387	388	389	390	391	392	393	394	395	396	397	398	399	400	401	402	403	404	405	406	407	408	409	410	411	412	413	414	415	416	417	418	419	420	421	422	423	424	425	426	427	428	429	430	431	432	433	434	435	436	437	438	439	440	441	442	443	444	445	446	447	448	449	450	451	452	453	454	455	456	457	458	459	460	461	462	463	464	465	466	467	468	469	470	471	472	473	474	475	476	477	478	479	480	481	482	483	484	485	486	487	488	489	490	491	492	493	494	495	496	497	498	499	500	501	502	503	504	505	506	507	508	509	510	511	512	513	514	515	516	517	518	519	520	521	522	523	524	525	526	527	528	529	530	531	532	533	534	535	536	537	538	539	540	541	542	543	544	545	546	547	548	549	550	551	552	553	554	555	556	557	558	559	560	561	562	563	564	565	566	567	568	569	570	571	572	573	574	575	576	577	578	579	580	581	582	583	584	585	586	587	588	589	590	591	592	593	594	595	596	597	598	599	600	601	602	603	604	605	606	607	608	609	610	611	612	613	614	615	616	617	618	619	620	621	622	623	624	625	626	627	628	629	630	631	632	633	634	635	636	637	638	639	640	641	642	643	644	645	646	647	648	649	650	651	652	653	654	655	656	657	658	659	660	661	662	663	664	665	666	667	668	669	670	671	672	673	674	675	676	677	678	679	680	681	682	683	684	685	686	687	688	689	690	691	692	693	694	695	696	697	698	699	700	701	702	703	704	705	706	707	708	709	710	711	712	713	714	715	716	717	718	719	720	721	722	723	724	725	726	727	728	729	730	731	732	733	734	735	736	737	738	739	740	741	742	743	744	745	746	747	748	749	750	751	752	753	754	755	756	757	758	759	760	761	762	763	764	765	766	767	768	769	770	771	772	773	774	775	776	777	778	779	780	781	782	783	784	785	786	787	788	789	790	791	792	793	794	795	796	797	798	799	800	801	802	803	804	805	806	807	808	809	810	811	812	813	814	815	816	817	818	819	820	821	822	823	824	825	826	827	828	829	830	831	832	833	834	835	836	837	838	839	840	841	842	843	844	845	846	847	848	849	850	851	852	853	854	855	856	857	858	859	860	861	862	863	864	865	866	867	868	869	870	871	872	873	874	875	876	877	878	879	880	881	882	883	884	885	886	887	888	889	890	891	892	893	894	895	896	897	898	899	900	901	902	903	904	905	906	907	908	909	910	911	912	913	914	915	916	917	918	919	920	921	922	923	924	925	926	927	928	929	930	931	932	933	934	935	936	937	938	939	940	941	942	943	944	945	946	947	948	949	950	951	952	953	954	955	956	957	958	959	960	961	962	963	964	965	966	967	968	969	970	971	972	973	974	975	976	977	978	979	980	981	982	983	984	985	986	987	988	989	990	991	992	993	994	995	996	997	998	999	1000	1001	1002	1003	1004	1005	1006	1007	1008	1009	1010	1011	1012	1013	1014	1015	1016	1017	1018	1019	1020	1021	1022	1023	1024	1025	1026	1027	1028	1029	1030	1031	1032	1033	1034	1035	1036	1037	1038	1039	1040	1041	1042	1043	1044	1045	1046	1047	1048	1049	1050	1051	1052	1053	1054	1055	1056	1057	1058	1059	1060	1061	1062	1063	1064	1065	1066	1067	1068	1069	1070	1071	1072	1073	1074	1075	1076	1077	1078	1079	1080	1081	1082	1083	1084	1085	1086	1087	1088	1089	1090	1091	1092	1093	1094	1095	1096	1097	1098	1099	1100	1101	1102	1103	1104	1105	1106	1107	1108	1109	1110	1111	1112	1113	1114	1115	1116	1117	1118	1119	1120	1121	1122	1123	1124	1125	1126	1127	1128	1129	1130	1131	1132	1133	1134	1135	1136	1137	1138	1139	1140	1141	1142	1143	1144	1145	1146	1147	1148	1149	1150	1151	1152	1153	1154	1155	1156	1157	1158	1159	1160	1161	1162	1163	1164	1165	1166	1167	1168	1169	1170	1171	1172	1173	1174	1175	1176	1177	1178	1179	1180	1181	1182	1183	1184	1185	1186	1187	1188	1189	1190	1191	1192	1193	1194	1195	1196	1197	1198	1199	1200	1201	1202	1203	1204	1205	1206	1207	1208	1209	1210	1211	1212	1213	1214	1215	1216	1217	1218	1219	1220	1221	12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DISTILLATION CURVES.

These curves were drawn using temperatures on the y-axis and total percentage distillation on the x-axis.

Comparing curves of fraction A of Run 1 and 2, the boiling range of the fraction has been slightly increased above 250⁰ C, which would indicate that a larger portion of oil was obtained in Run 2, between 250 and 390 than in Run 1. From this we would expect a slight increase of anthracene salts in fraction 300-390⁰ C which is shown to be the case when comparing the anthracene yield in the table. This was due to the vacuum which effected the vapor pressure of the tar.

Comparing curves of fraction A of Run 1 and 3, we find that the difference of the curves is more marked than in the previous case. The boiling range has been increased between the temperatures of 310 and 390, showing that oils of higher boiling range must have been obtained in this run. We would expect an increase in anthracene salts in Run 3 over Run 2. Anthracene salts yield shown in table bear out this conclusion.

In comparing Run 1 and 4, fraction A, we note that the difference pointed out in the above case is not as great as Run 3.

Continuing the comparison, Run 5 shows a greater difference than in Run 3.

Curve of Run 6, shows the smallest difference so far noted, although the lower end shows a greater quantity of lower boiling oils.

Curve of Run 7, shows a difference almost equal to that of Run 4.

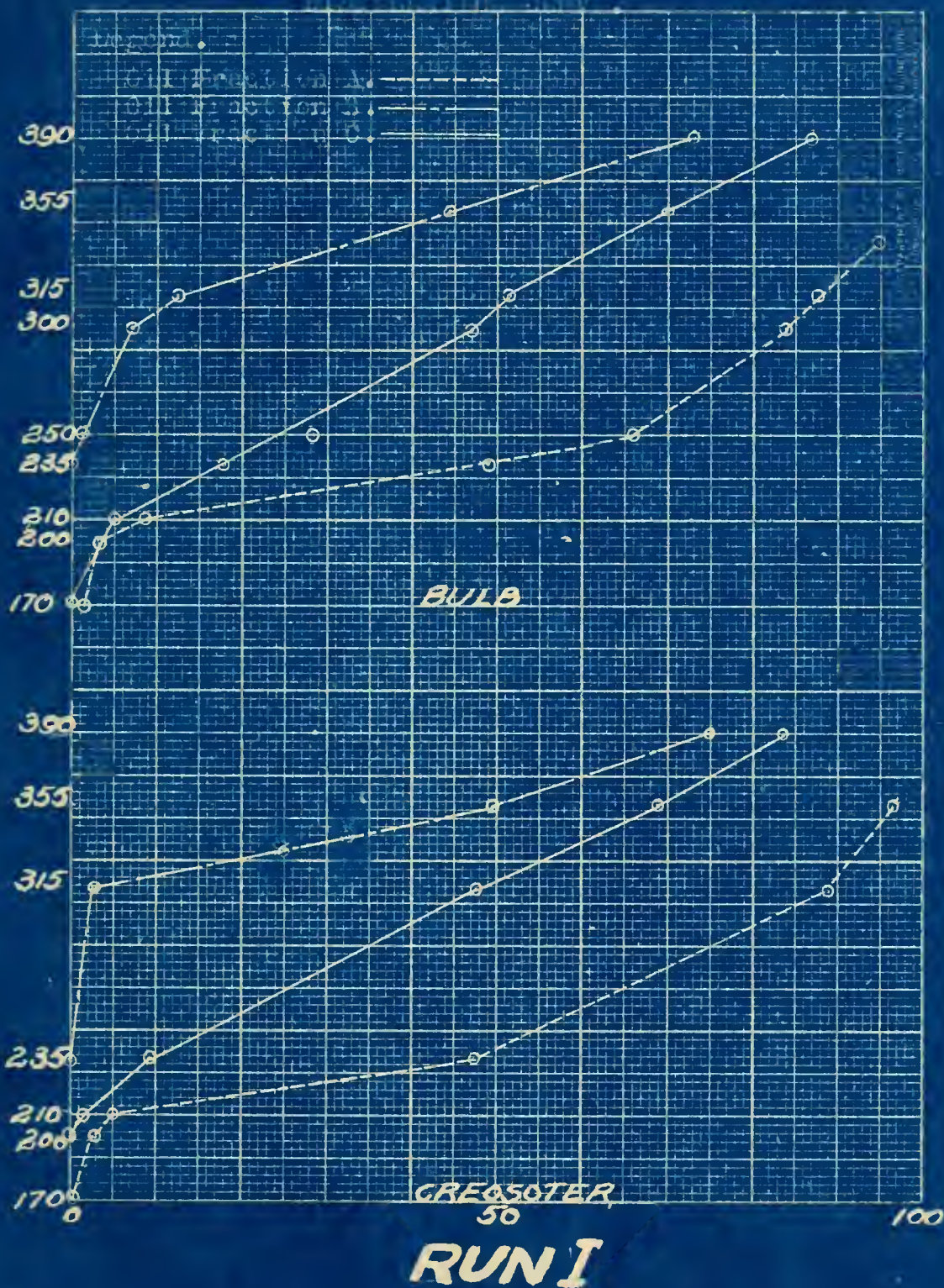
Comparing curves of Run 1 and 4, fraction B, we note that they are approximately the same.

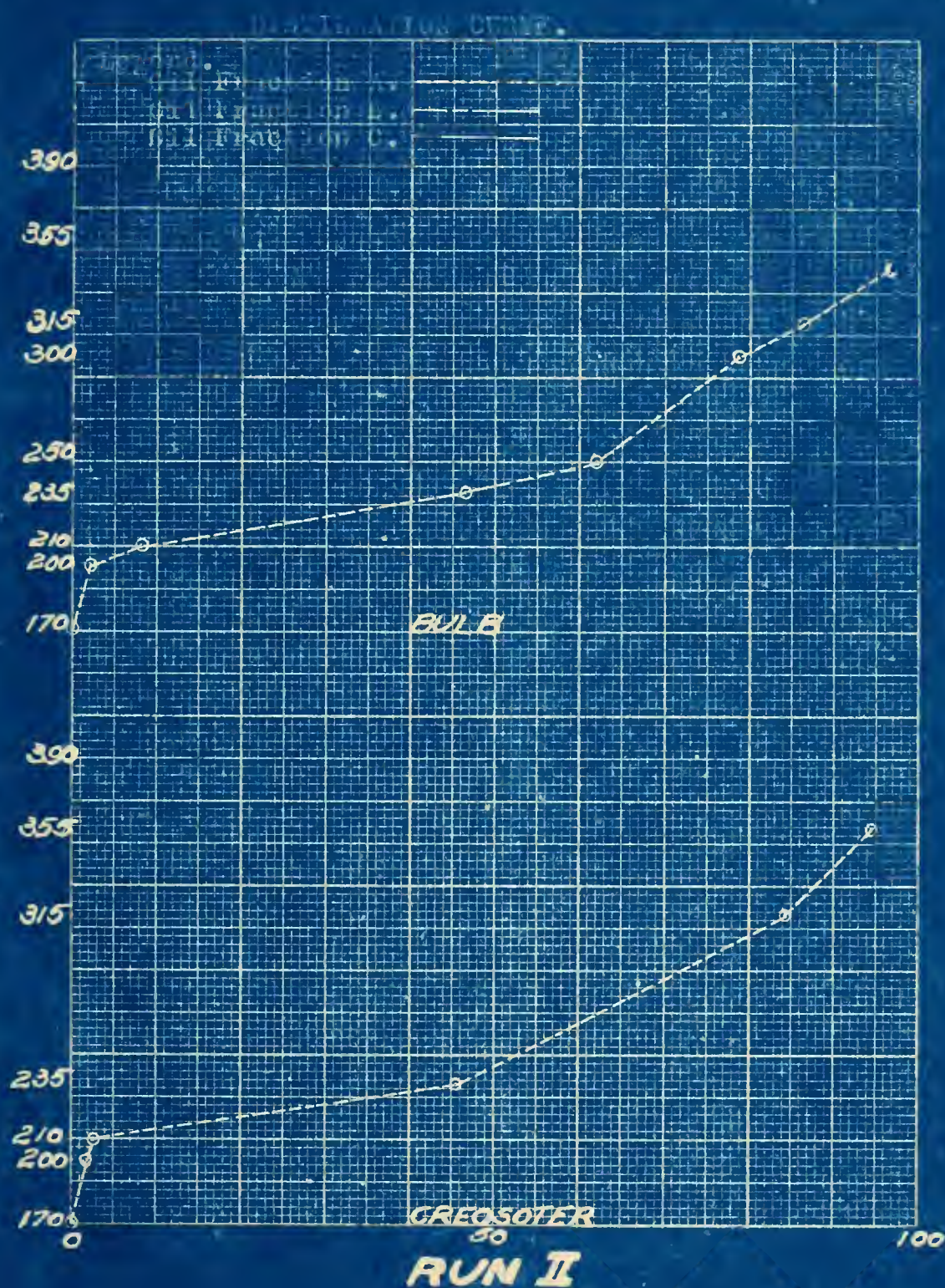
Curve B, of Run 5, indicates to be a higher boiling range oil and one would expect to find more anthracene salts. Table shows increase of anthracene salt yield over Run 1.

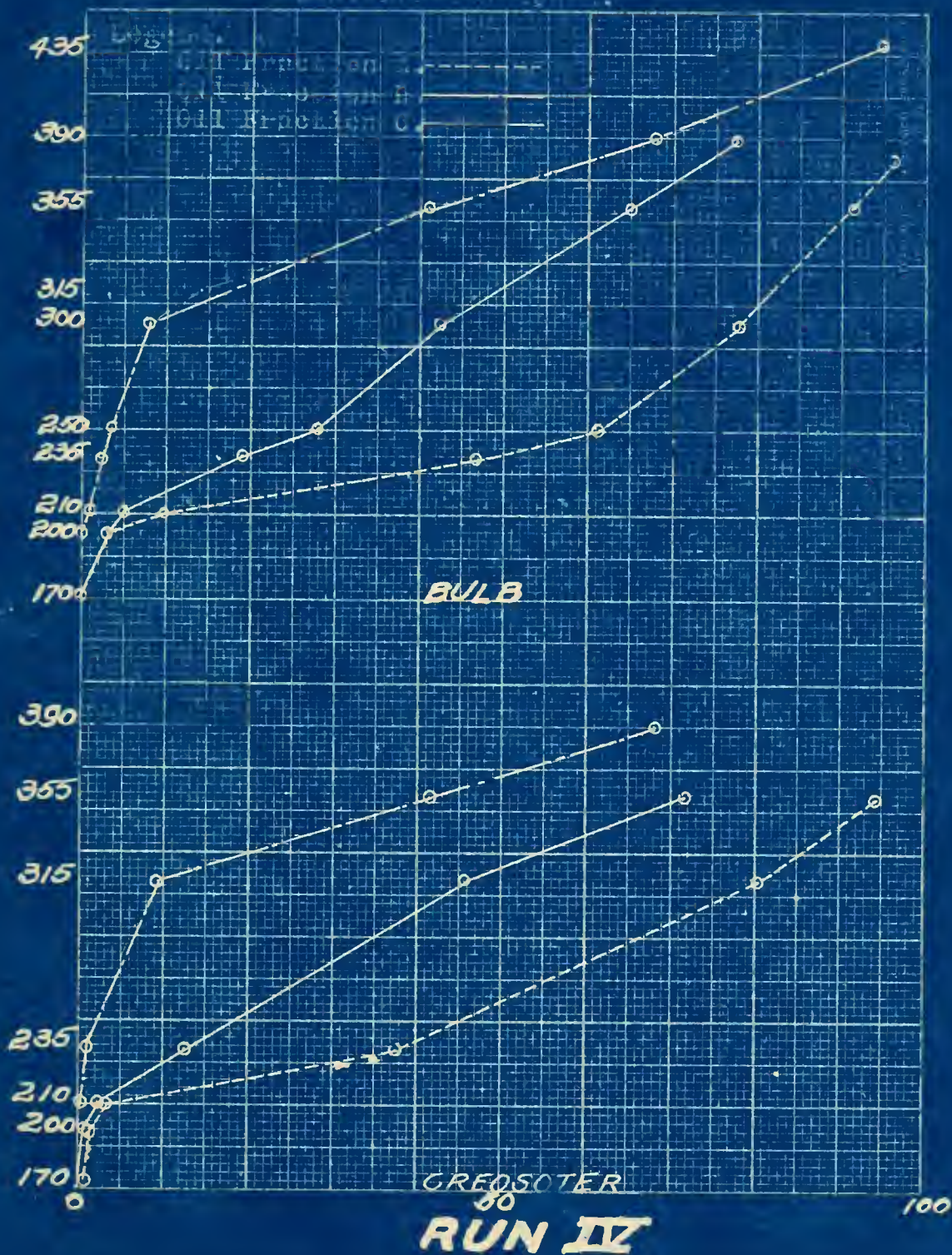
Curve B of Run 6 points out that the oil is of a lower boiling range. Smaller yield of anthracene salts would be expected in this oil. Results showed in table bear out this view.

Curve B of Run 7, shows that the boiling range is considerably lower than in Run 1 up to

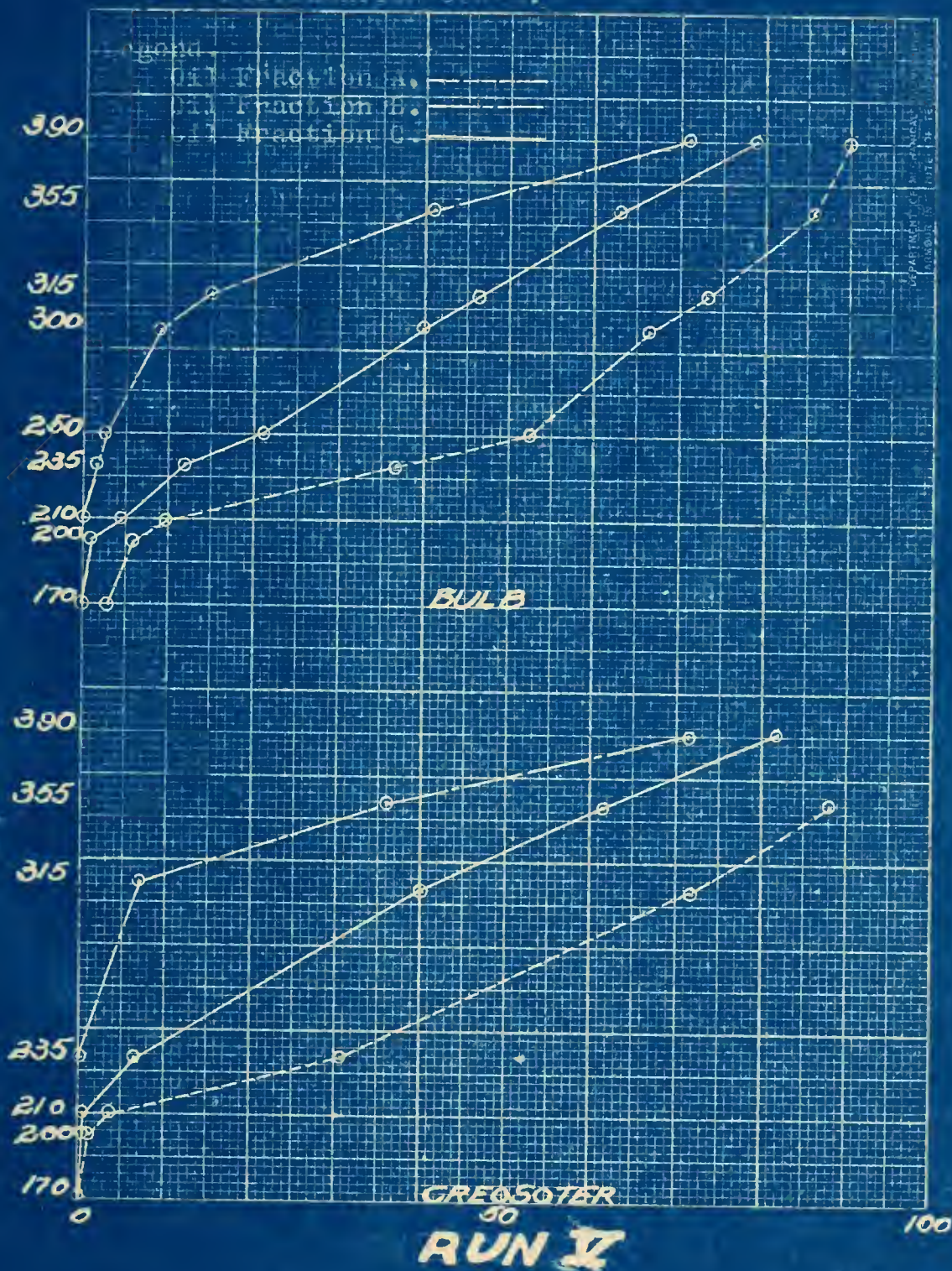
315° C and then becomes an oil of a higher boiling range. This indicates a mixture of lower and higher boiling oils. One would expect from such an oil a low yield of anthracene salts. Anthracene content of this fraction of these runs, (shown in table) bears this out.

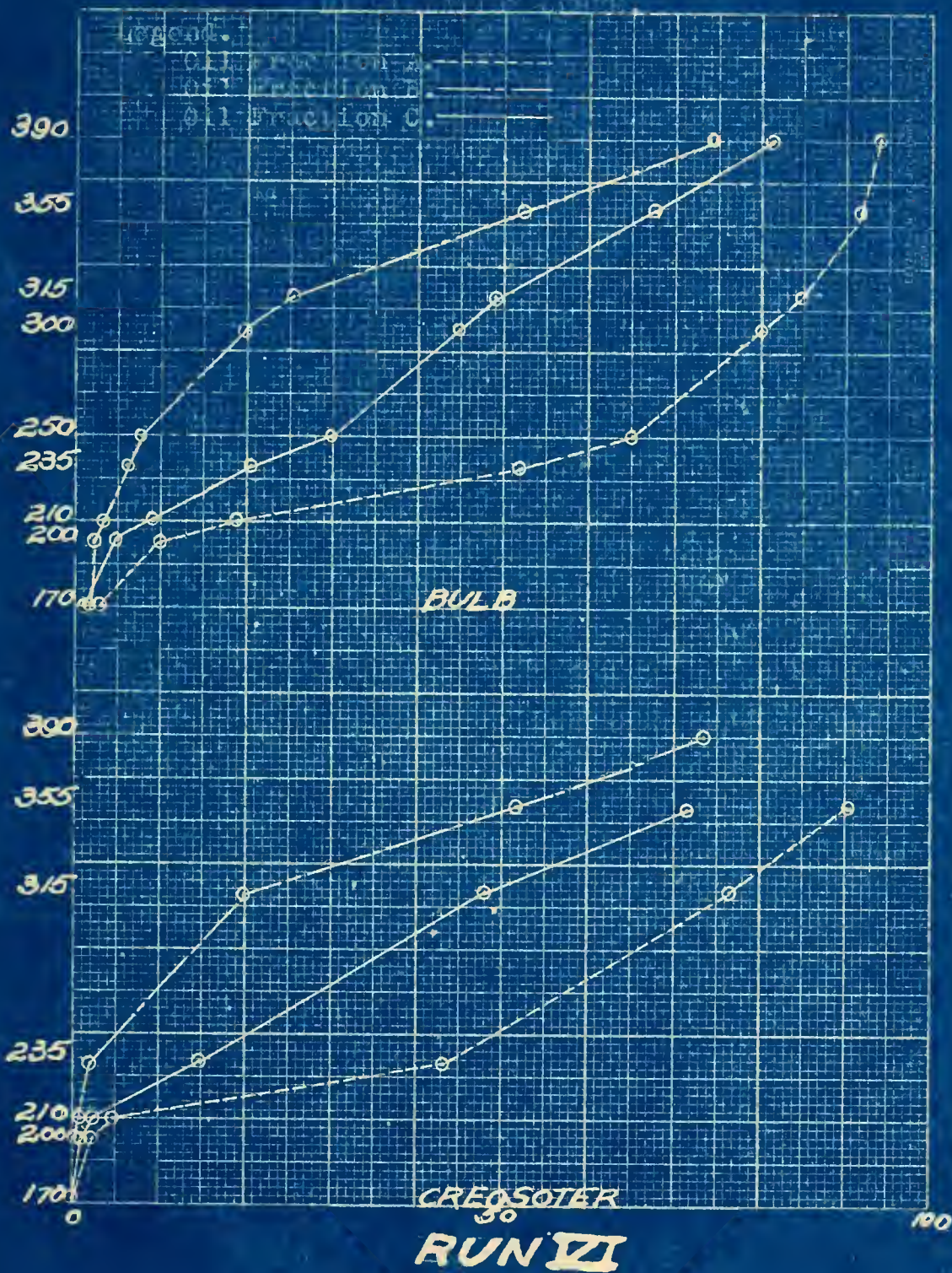


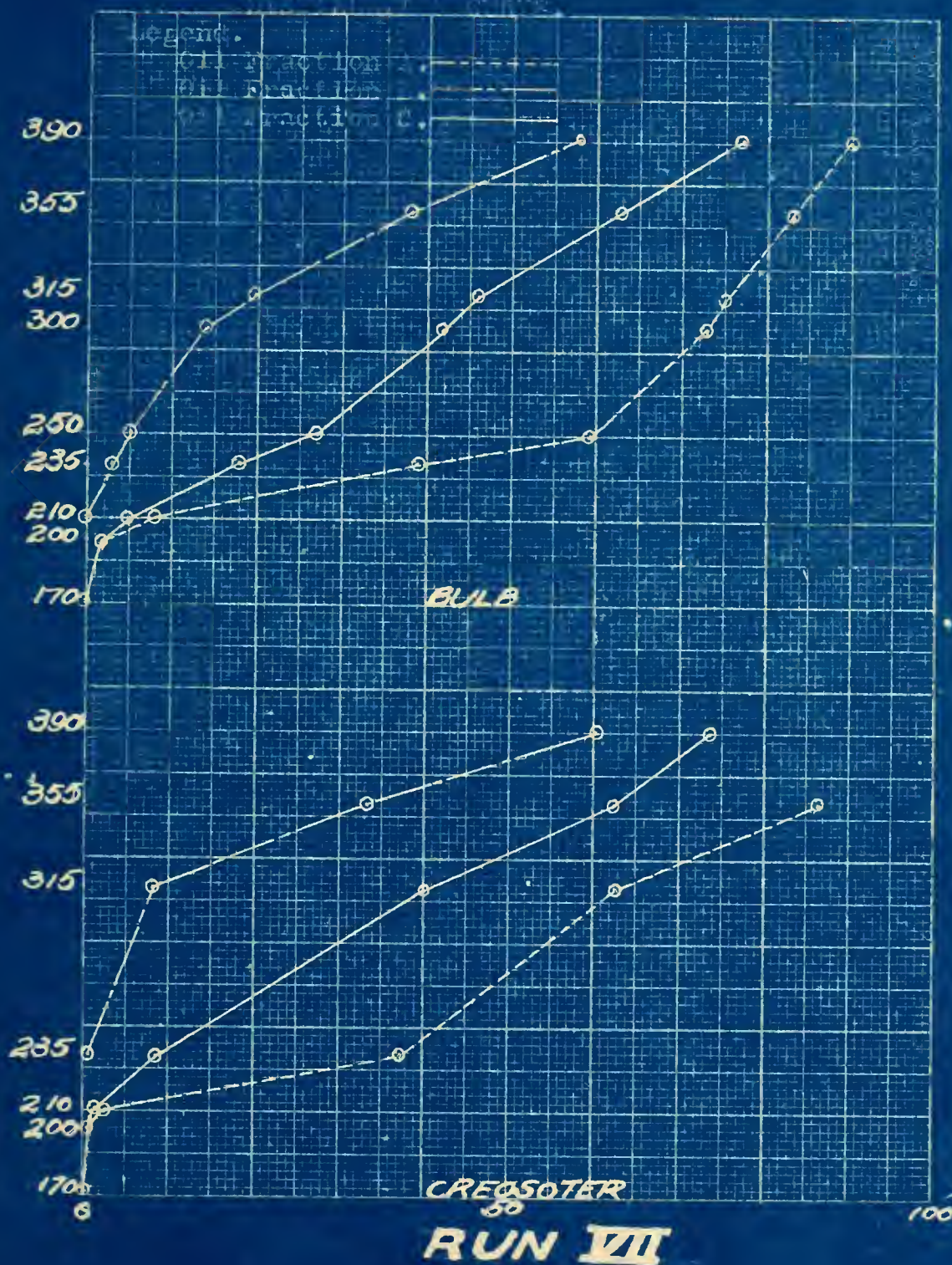




DISTILLATION CURVE.







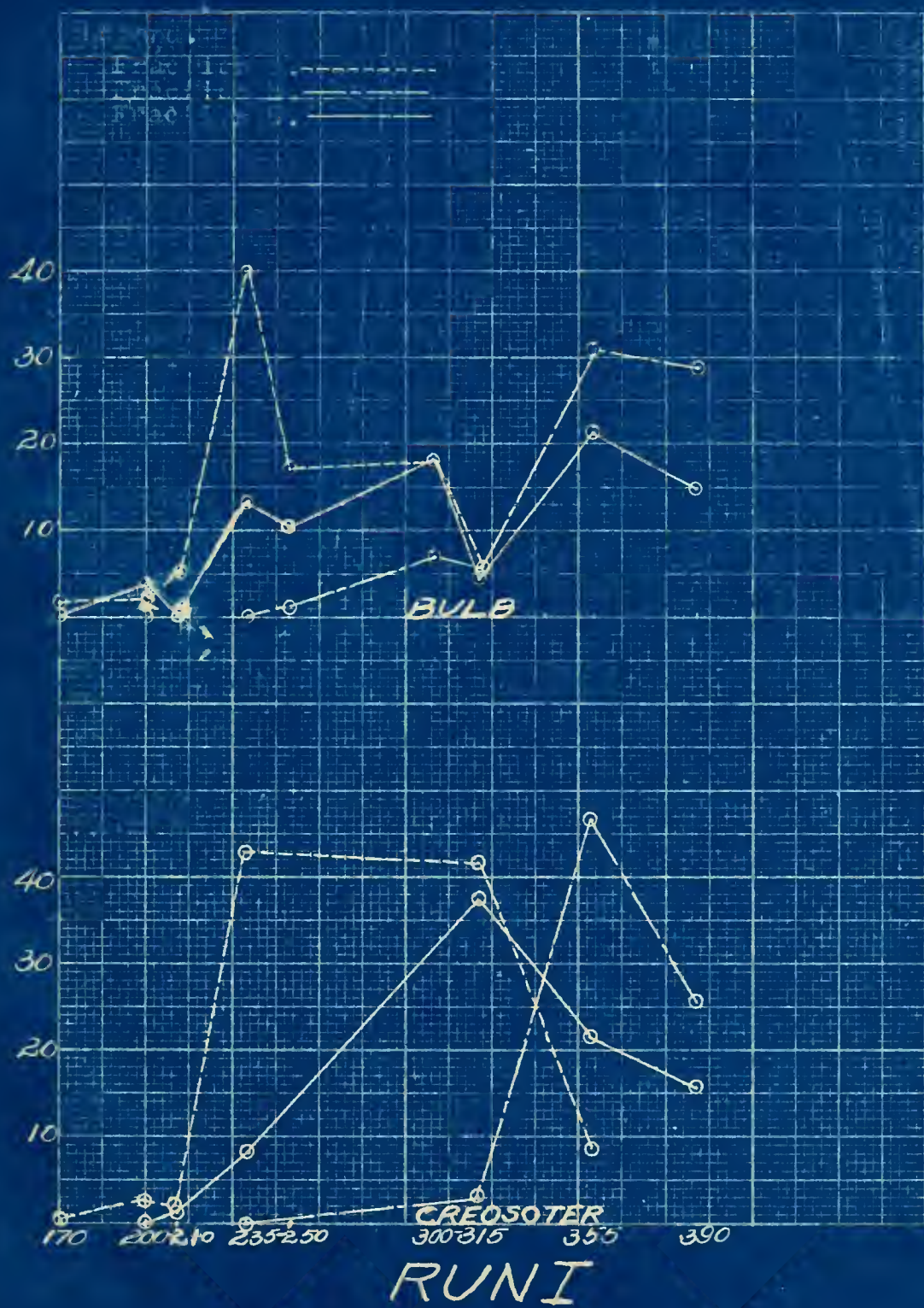
PER CENT OF OIL PER TEMPERATURE DIFFERENTIAL.

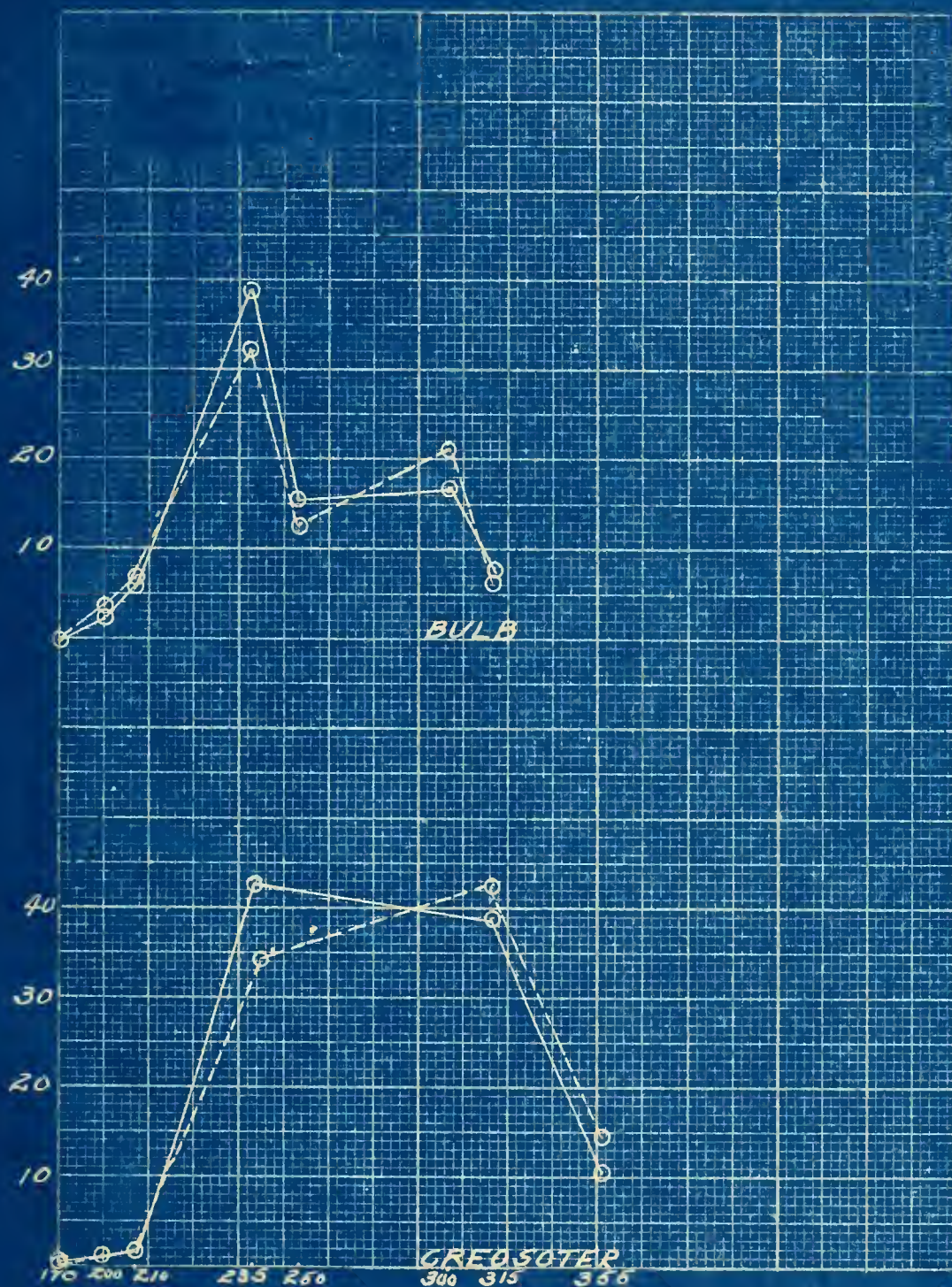
These curves were made using the x-axis for temperatures and the y-axis as the difference between per centage of distillate for each temperature differential.

They show the same differences as the distillation curves.

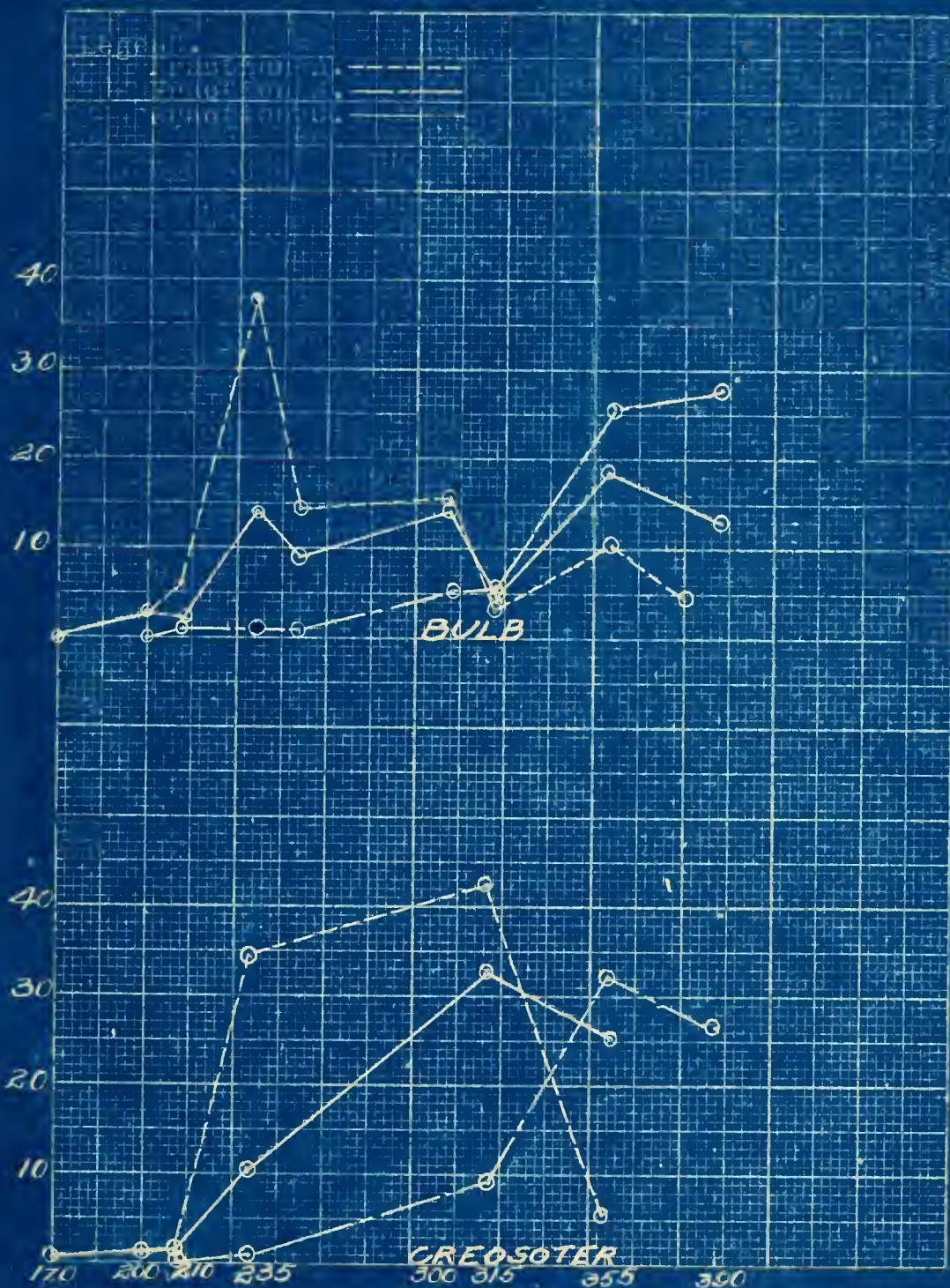
In comparing the per cent of oil per temperature differential curves of Fraction A, it is to be noted that the portion of curves up to 250⁰ C indicate that the naphthalene content of the oils obtained gradually decrease from Run No.1 to inclusive Run 5. The curve of Run 5 indicates the lowest naphthalene content for the oils of Fraction A. The naphthalene content of Run 6 is increased above that of Run 5 but it is lower than that of Run 4 and Run 7. The curve of Run 7 indicates that the naphthalene content is increased over that of Run 6. Multiplying the No. of c.c. of the fraction A with the per cent of naphthalene salts obtained we obtain for Run No.1 426 grms; for Run 2, 407 grms; for Run 3, 405

grms; for Run 4, 401 grms; for Run 5, 307 grms; for Run 6, 339 grms; and for Run 7, 368 grms. The distillation curves and the % of naphthalene salts obtained indicate that the naphthalene fraction was not affected by the hydrogen when the catalytic agent was used in the contact chamber. That the naphthalene fraction was affected when the catalytic agent aluminum chloride was used in the tar.



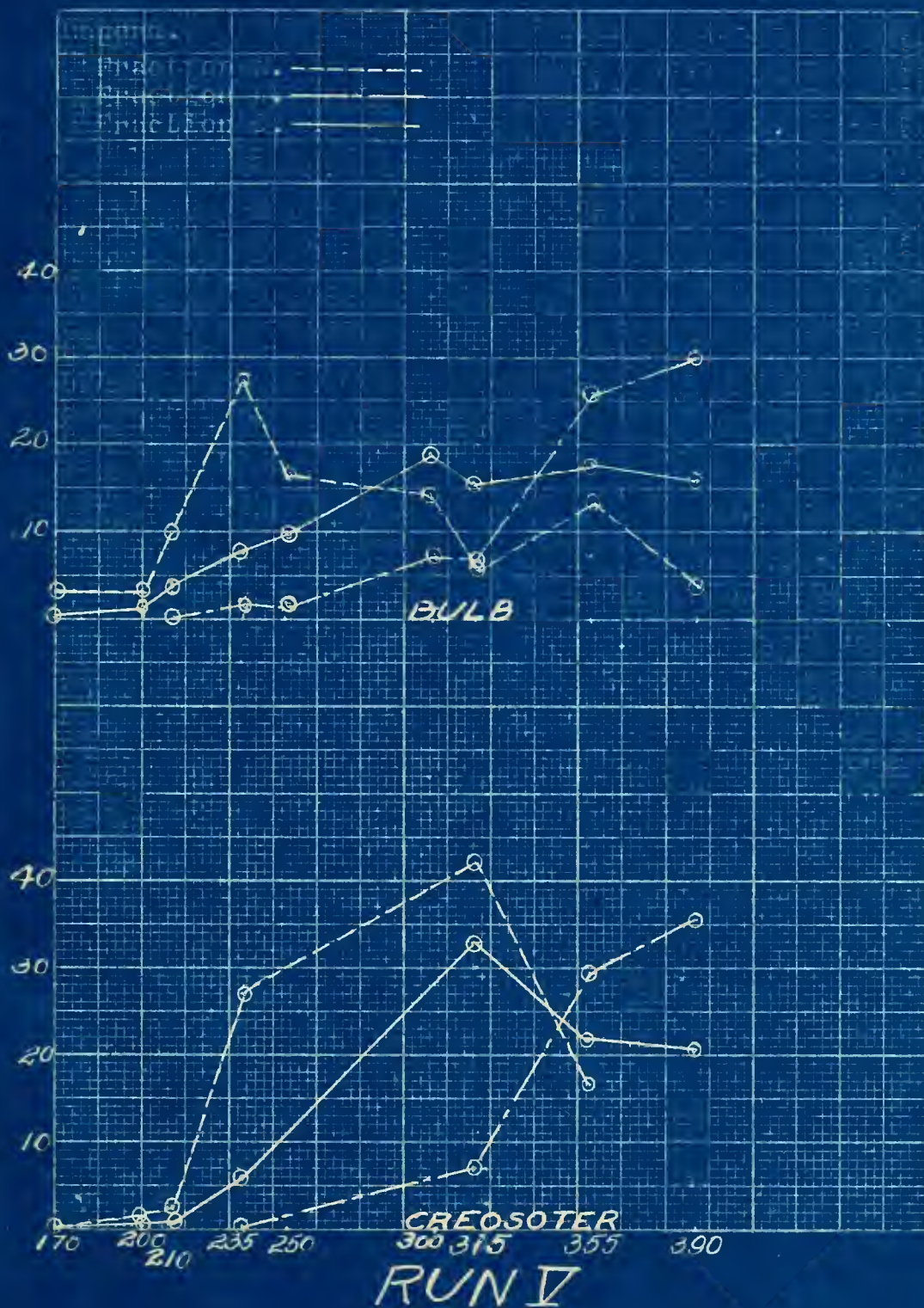


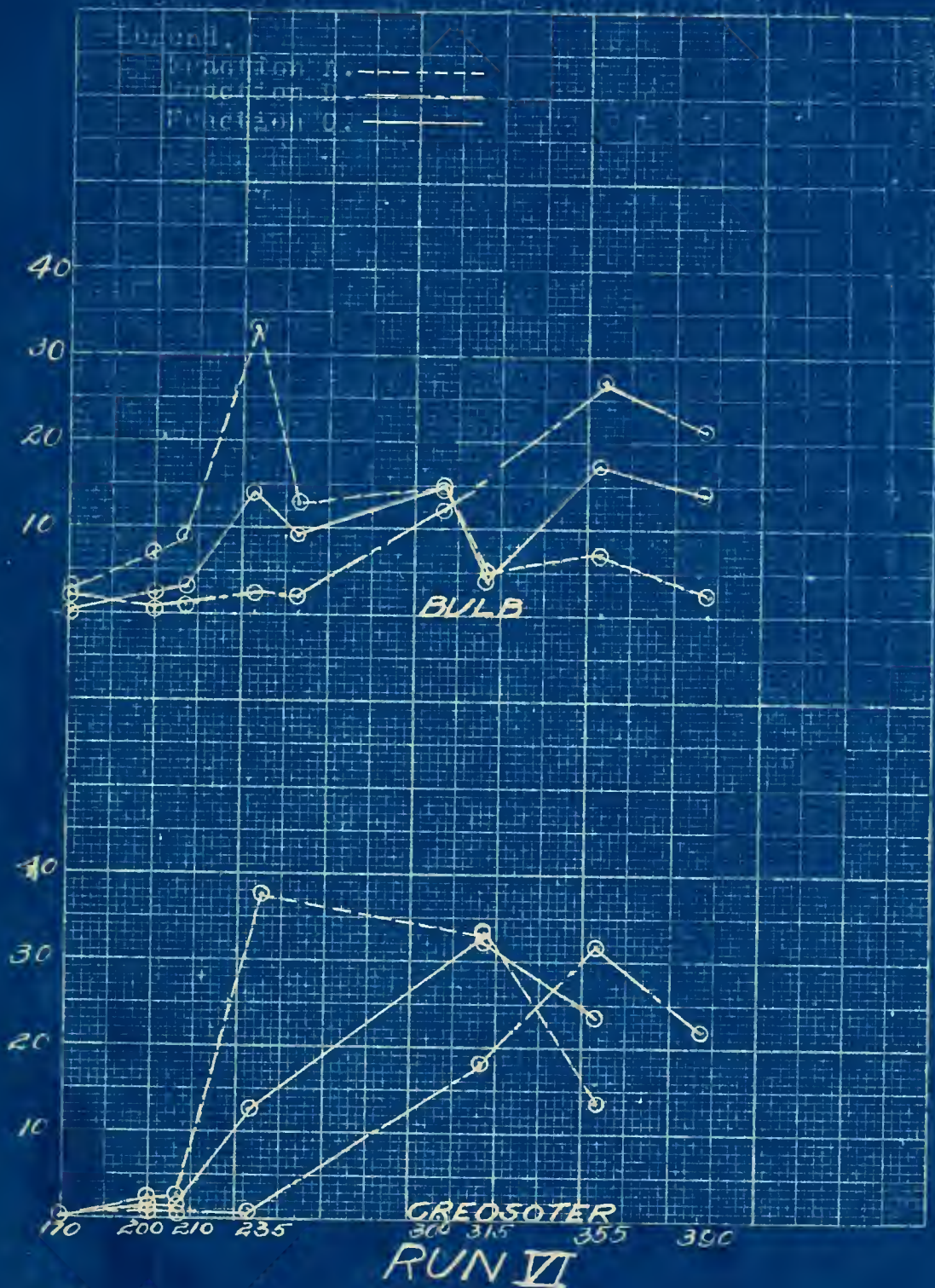
RUN II —
 RUN III ---- FRACTION A.

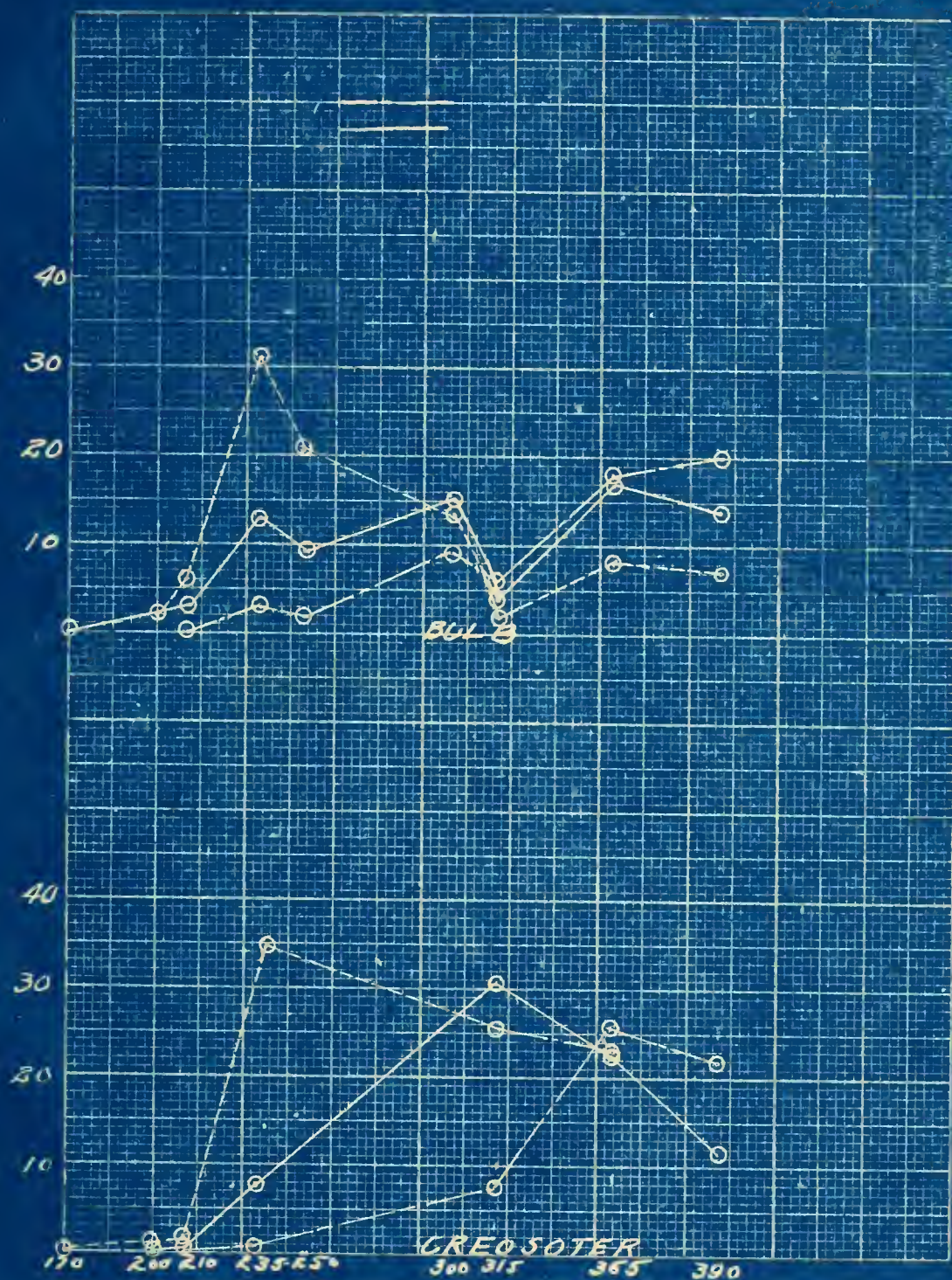


RUN IV

FOR 2000 TO 1000 PSI. (1000 PSI. TO 1000 PSI.)







RUN VII



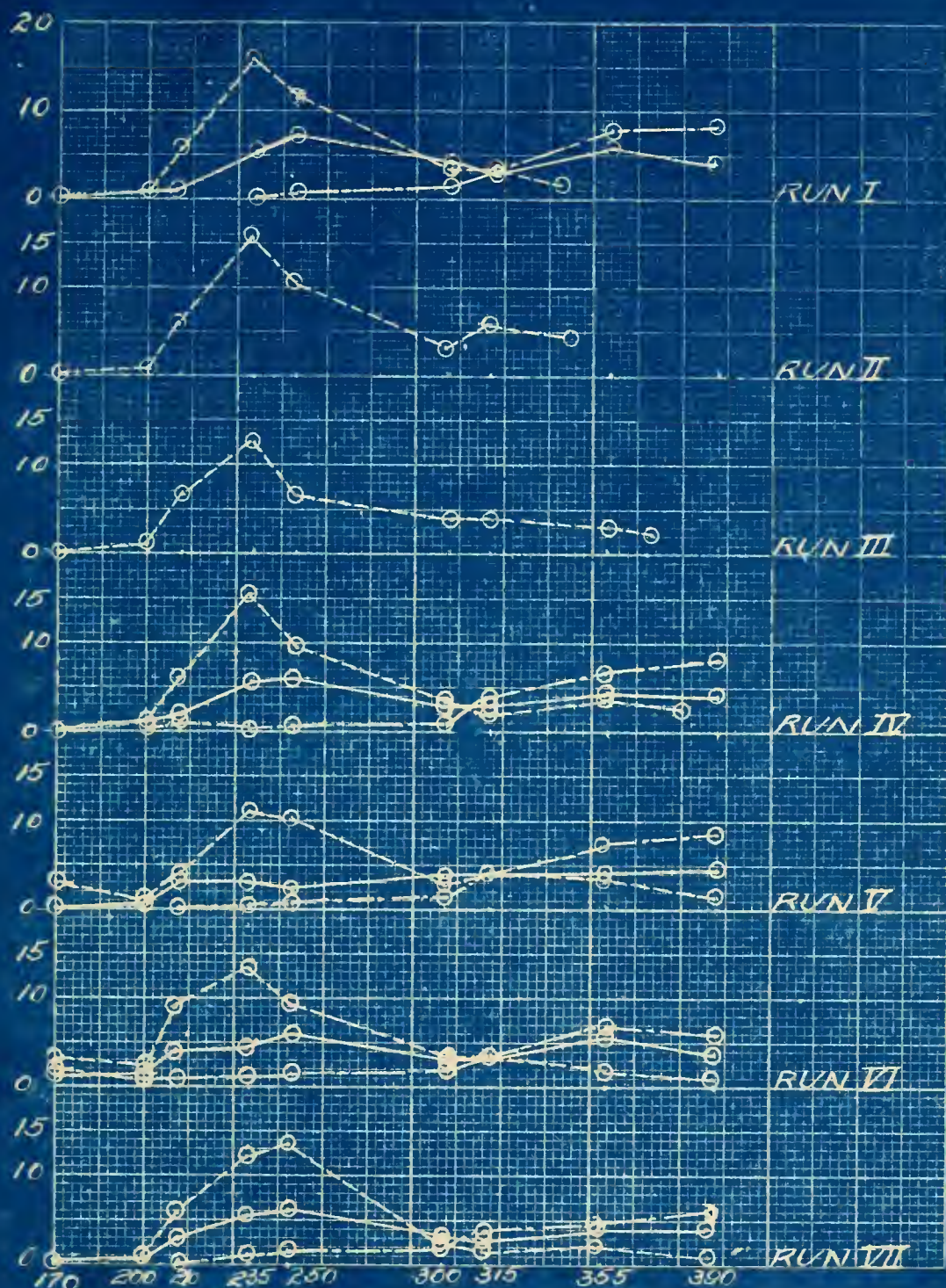
PER CENT OF DISTILLATE PER UNIT TEMPERATURE DIFFERENTIAL CURVES.

The bulb distillation results of the seven runs were used in the construction of this graph. The percentage obtained for each temperature differential were divided by the temperature differential and multiplied by ten and plotted on the y-axis of the graph. Temperatures of distillation were plotted on x-axis. For illustration Run 1, fraction A, bulb distillation is

TEMPERATURE DIFFERENTIAL	PER CENT OF OIL PER TEMPERATURE DIFFERENTIAL	PER CENT OF OIL PER UNIT TEMPERATURE DIFFERENTIAL.
170-1.5		
200-3.5	30	2.0
210-9.0	10	5.5
235-49.0	25	40.0
250-66.	15	17.0
300-84	50	18.0
315-89.5	15	5.5
340-95	25	5.5
		0.66
		5.5
		16.0
		11.3
		3.6
		3.7
		2.20

These curves show where the greatest amount of oil is distilled per degree for each temperature differential. It further shows that the percentage distillation of fraction C is not an arithmetic mean of fraction A and B. The per-

centage distillation of a composite cannot
therefore be foretold from the distillation
of its components.



LEGEND: FRACTION A. --- FRACTION B. --- FRACTION C.

CONCLUSIONS:

1. The marked increase of anthracene salts when catalytic agents and hydrogen were used is to be especially noted. The reason is probably that polymerization has taken place in the "contact chamber".

2. From tests of oils, it is evident that the per cent distillation per temperature differential of the composite is not the arithmetic mean of its components. This leads to the conclusion that by a reasonable number of distillations, a Coal Tar product such as anthracene could not be concentrated to any high degree of purity.

3. Due to the deposition of carbon on the catalytic material and the presence of luminous gases coming off with the hydrogen one would be led to conclude that dehydrogenation has taken place to some extent.

4. If coal tar is to be hydrogenated commercially for the production of anthracene salts and increased oil yield per gal of tar, we would propose an intermittant operation with

a large "catalytic" area; operating half of the catalytic area for hydrogenation while the other half be flushed with an inert gas to replace the hydrogen and the hydrocarbons and the deposited carbon removed by combustion with air or pure oxygen.

5. The yield of naphthalene from the tar is not affected when the tar is distilled under vacuum or when the hydrogen and the vapors of distillation are passed over the catalytic agent in the contact chamber. The yield of naphthalene is decreased when distillation with hydrogen is made with aluminum chloride in tar. The decrease is largest when distillation is made with aluminum chloride in tar and passing the vapors of distillation and the hydrogen over reduced aluminum chloride in the contact chamber.

REFERENCES.

The methods of analysis used to obtain results in the table are those published by J.W. Weiss in The Journal of Industry and Engineering Chemistry, Vol. 10, No.9, pp. 732-738; Vol. 10, No.10, pp. 817-823; Vol. 10, No.11, pp. 911-916 and Vol. 10, No. 12, pp. 1006-1012.

OTHER REFERENCES:

"The Pyrogenesis of Hydrocarbons" Part 1, Historical and Bibliographical, By E. Lawson Lomax, Journal of Industrial and Engineering Chemistry, Vol. 9, No.9, pp. 879 to 902.

